[Second Edition.]

PATENT SPECIFICATION

Application Date: April 3, 1923. No. 30,872/23.

" " Dec. 8, 1923. No. 30,873/23.

One Complete Left: Dec. 8, 1923.

Complete Accepted: July 3, 1924.

218,563

PROVISIONAL SPECIFICATION.

No. 30,872, A.D. 1923.

Improvements in and relating to the Manufacture of Alkali Metals.

I, (Dr.) THOMAS EWAN, a British subject, of 86, Shuna Street, Maryhill, Glasgow, do hereby declare the nature of this invention to be as follows:—

This invention relates to an improvement in the manufacture of alkali metals and especially to the manufacture of sodium.

According to my invention I electrolyse an aqueous solution of an alkali salt with a mercury cathode in a divided cell and I remove the alkali metal from the amalgam by the aid of liquid anhydrous ammonia. My invention further consists in the process of separating sodium (or other alkali metal) as such from amalgam especially from a very dilute amalgam by the aid of an inert electrolytic solution at low temperatures *i.e.* at temperatures much lower than 100° C. The preferred range is from $-30°$ to $+30°$ C. which is far below those which can be obtained by the use of fused salts. The solvent I prefer to use is liquid anhydrous ammonia but the invention is not limited thereto as other liquids may be used provided that they yield electrolytic solutions inert to sodium, that is, the solutions must not react with the sodium to an extent which will prevent isolation of the sodium therefrom. Liquid methylamine is an example of an alternative solvent. Fused salts are unsuitable because they are too hot. Many organic liquids are excluded because they react with sodium.

The electrolyte may be any sodium (or alkali-metal) salt which is soluble in the solvent and which is inert (in the above-defined sense) to sodium (or other alkali metal) for example sodium cyanide.

The invention includes the use of certain solutions in which sodium is substantially insoluble but which can be used to remove sodium from the amalgam. The sodium can thus be separated as a solid direct but a solution in the liquefied gas may be drawn off continuously to yield metallic sodium by evaporation when the whole process of isolation of the metal may be made continuous.

The invention also includes a new electrolyte, namely, concentrated solution of a metallic cyanide in liquid anhydrous ammonia.

The invention will now be described in detail with reference to the isolation of metallic sodium by the aid of liquid anhydrous ammonia when certain further features of novelty will be apparent.

It is known that sodium dissolves in anhydrous ammonia, the more dilute solutions being dark blue and the more concentrated ones possessing metallic lustre resembling copper. It is also known that at low temperatures these solutions separate into two liquid phases of different composition. This separation occurs at somewhat higher temperatures when sodium chloride is added to the solution of sodium in ammonia. (C. A. Kraus, Journal of the American Chemical Society Vol. 29 p. 1557, 1907).

My experiments lead to the conclusion that solutions of sodium salts in liquid anhydrous ammonia may be divided into three groups:—

(i) Those in which sodium dissolves; the solution may or may not consist of two phases.

(ii) Those in which sodium is insoluble and with which it co-exists in the form of a solution of sodium in ammonia free from salts.

(iii) Those in which sodium is insoluble and with which it co-exists in the form of solid metal.

When a readily soluble sodium salt is

added in increasing quantities to a solution of sodium in ammonia a point can be reached at which the solution separates into two distinct layers, the lower one consists of a colourless solution of the sodium salt in ammonia and the upper one containing the whole of the sodium dissolved in ammonia and so free from the salt that I have failed to detect its presence. Further increase of the concentration of the salt increases the concentration of the sodium solution in equilibrium with it until saturation is reached, beyond this point solid sodium separates out.

Solutions belonging to the first group (those in which sodium dissolves appreciably) are of little value in the manufacture of sodium because the metal which is formed at the cathode dissolves in the electrolyte and is reabsorbed therefrom by the mercury at the anode. Further any sodium which escapes reabsorption can only be removed from the cell together with the dissolved sodium salt from which it must be separated, in a subsequent operation, a very undesirable complication of the process. The more nearly the solutions approach the border line which divides them from group two the less do these objections hold good.

The solutions of the second group (those from which sodium separates completely in the form of a solution in ammonia) are very suitable electrolytes for the purposes of this invention. The sodium solutions float on the surface of the salt solutions and are thereby effectively kept out of contact with the anode mercury. Being liquid, they are readily removed from the electrolytic cell, and the sodium is easily recovered in a pure condition by evaporating off the ammonia.

Solutions of the third group are also suitable although the deposition of solid sodium, with its tendency to form dendritic crystalline growths and the removal of the solid metal from a cell, which, on account of the volatile nature of the solution is necessarily closed gas-tight present difficulties which are not met with when sodium is produced in a liquid form. The number of sodium salts which yield suitable electrolytic solutions is limited. The majority are either insoluble or so sparingly soluble in ammonia that only solutions of the first group are obtainable. Sodium chloride and bromide for example yield solutions of this first group only.

Other salts which are sufficiently soluble cannot be used because they are not inert, *i.e.* they react with sodium; sodium nitrate and thio cyanate are examples.

Sodium cyanide and sodium iodide are sufficiently soluble to yield solutions of groups two and three, they do not react with sodium in presence of liquid ammonia and the solutions are good conductors of electricity but I prefer the cyanide since there is a danger of forming explosive products in certain cases when using the iodide.

In practice very satisfactory results are obtained by the use of an electrolyte containing 33 parts of sodium cyanide to 67 parts of ammonia, which yields a solution of sodium in ammonia containing 15 to 16 *per cent.* of the metal. A solution containing equal weights of sodium cyanide and ammonia yields solid sodium.

The sodium amalgam may be of any strength, but the very dilute, liquid amalgams which are easily obtained are preferred. In using these the current density at the anode must be proportioned to the sodium content of the amalgam; if too high current density is employed mercury dissolves as well as sodium and the production of sodium is diminished. So far my experiments have indicated that an anodic current density of 3 or 4 amperes per square centimetre for each 1 *per cent.* of sodium in the amalgam is safe. The current density at the cathode may be quite small as compared with processes in which fused salts are electrolysed.

The cathode is suitably made of iron, but many other materials which conduct electricity and are not acted on by the substances present in the cell are available. Sodium itself is suitable if the solid metal is being made. Metals which accelerate the normally very slow reaction between sodium and ammonia to form sodium amide should be avoided. Metals such as lead which form alloys with sodium should be avoided since the sodium is to be isolated as such or after evaporation of the solution. It will be seen that the electrolysis is not for the purpose of decompositing the dissolved sodium salt but for removing the metallic sodium from the amalgam.

A very material technical advantage of the process lies in the low temperatures at which it works. Heat added to the electrolyte by the passage of the electric current through it or from any other source first raises the temperature of the electrolyte to its boiling point under the pressure which is maintained in the cell and then causes ammonia to evaporate; the temperature is thus restricted by the boiling point of the electrolyte. The evaporated ammonia is of course, condensed and returned to the cell, excessive

boiling is conveniently prevented by removing heat by means of an external cooling agent. For example, if the cell is jacketed with liquid ammonia boiling at atmospheric pressure the temperature is maintained but little above $-33°$ and the pressure within the cell may be one atmosphere. If, on the other hand, cold water is used as the cooling agent the temperature of the cell will rise to 15° of 20° and the pressure to 100 to 150 lbs. per square inch. Very low temperatures cannot be used, partly on grounds of economy, partly because the electrolyte may solidify more or less completely. For example certain solutions of sodium cyanide in ammonia freeze at $-31°$. The range of temperatures within which the process can be worked is therefore approximately from $-30°$ to $+30°$. It is this low temperature range which makes the use of mercury possible since losses by vaporisation are practically suppressed.

Dated this 7th day of December, 1923.

W. P. THOMPSON & Co.,
12, Church Street, Liverpool,
Chartered & Registered Patent Agents.

PROVISIONAL SPECIFICATION.

No. 30,873, A.D. 1923.

Improvements in and relating to the Manufacture of Alkali Metals.

I, (Dr.) THOMAS EWAN, a British subject, of 86, Shuna Street, Maryhill, Glasgow, do hereby declare the nature of this invention to be as follows:—

This invention relates to an improvement in the manufacture of alkali metals.

According to my invention I electrolyse an aqueous solution of an alkali salt with a mercury cathode in a divided cell and I remove the alkali metal from the amalgam by the aid of liquid anhydrous ammonia. My invention further consists in the process of separating sodium (or other alkali metal) as such from amalgam especially from a very dilute amalgam by using the amalgam as anode in the electrolysis of an inert electrolytic solution at low temperatures *i.e.* at temperatures much lower than 100° C. The preferred range is from $-30°$ C. to $+30°$ C. which is far below those which can be obtained by the use of fused salts. The solvent I prefer to use is liquid anhydrous ammonia but the invention is not limited thereto as other solvents may be used provided that they yield electrolytic solutions inert to sodium or other alkali metal, that is, the solutions must not react with the sodium or alkali metal to an extent which will prevent isolation of the metal therefrom. Liquid methylamine is an example of an alternative solvent. Many organic liquids are excluded because they react with sodium or alkali metal. Salts in the fused condition are unsuitable because their melting points are too high.

The electrolyte may be any sodium (or alkali-metal) salt which is soluble in the solvent and which is inert (in the above-defined sense) to sodium (or other alkali metal) for example, sodium (or other alkali metal) cyanide.

The invention includes the use of certain solutions in which alkali metals are substantially insoluble but which can be used to remove them from their amalgams. The metals can thus be separated as solids direct, but solutions in the liquefied gas may be drawn off continuously to yield the metals by evaporation. The whole process of isolation of the metals may be made continuous.

My experiments lead to the conclusion that solutions of alkali metal salts in liquid anhydrous ammonia may be divided into three groups:—

(1) Those in which the corresponding metal dissolves; the solution may or may not consist of two phases.

(11) Those in which the metal is insoluble or very sparingly soluble and with which it co-exists in the form of a solution of the metal in ammonia free or nearly free from salts.

(111) Those in which the metal is substantially insoluble and with which it co-exists in the solid form.

When a readily soluble sodium salt for example, is added in increasing quantities to a solution of sodium in ammonia a point can be reached at which the solution separates into two distinct layers, the lower one consisting of a solution of the sodium salt in ammonia containing mere traces of free sodium and the upper one containing substantially the whole of the sodium dissolved in ammonia and almost free from the salt. Further increase of the concentration of the salt increases the concentration of the sodium

solution in equilibrium with it until saturation is reached, beyond this point solid sodium separates out.

Solutions belonging to the first group (those in which the metal dissolves more or less readily) are of little value in the manufacture of the metals by my invention because the metal which is formed at the cathode dissolves more or less readily in the electrolyte and is reabsorbed therefrom by the mercury at the anode. Further any metal which escapes reabsorption can only be removed from the cell together with the dissolved salt from which it must be separated, in a subsequent operation, a very undesirable complication of the process. The more nearly the solutions approach the border line which divides them from group two, the less do these objections hold good. When using dilute solutions I prefer to use a diaphragm to hinder contact of dissolved sodium with mercury.

The solutions of the second group (those from which alkali metal separates almost completely in the form of a solution in ammonia) are very suitable electrolytes for the purposes of this invention. The metal solutions float on the surface of the salt solutions and are thereby effectively kept out of contact with the anode amalgam. Being liquid, they are readily removed from the electrolytic cell, and the metal is easily recovered in a pure condition by evaporating off the ammonia.

Solutions of the third group are also suitable although the desposition of solid metal, with its tendency to form dentritic crystalline growths and the removal of the solid metal from a cell, which, on account of the volatile nature of the solution is necessarily closed gas-tight present difficulties which are not met with when the metal is produced in the form of a liquid solution.

The number of sodium or alkali metal salts which yield suitable electrolytic solutions is limited. The majority are either insoluble or so sparingly soluble in ammonia that only solutions of the first group are obtainable. Sodium chloride and bromide for example, yield solutions of this first group only.

Other salts which are sufficiently soluble cannot be used because they are not inert, *i.e.* they react with sodium or other alkali metal the nitrates and thio cyanates being examples.

Sodium cyanide and sodium iodide are sufficiently soluble to yield solutions of groups two and three. They do not react with sodium in presence of liquid ammonia and the solutions are good conductors of electricity but I prefer the cyanide since there is a danger of forming explosive products in certain cases when using the iodide.

The alkali metal amalgam may be of any strength, but the very dilute, liquid amalgam which are easily obtained are preferred. In using these the current density at the anode must be proportioned to the alkali metal content of the amalgam and to its rate of movement; if too high current density is employed mercury dissolves as well as alkali metal and the production of the latter is diminished. So far my experiments have indicated that (with sodium amalgam for example) an anodic current density of 3 or 4 amperes per square centimetre for each 1 *per cent.* sodium in a stationary amalgam is safe but very much higher current densities can be employed if the amalgam is stirred or flows in a thin sheet; if these critical current densities are surpassed mercury dissolves, when the production of sodium may stop. The current density at the cathode may be quite small as compared with processes in which fused salts are electrolysed.

The cathode is suitably made of wrought iron or mild steel, but many other materials which conduct electricity and are not acted on by the substances present the cell are available, *e.g.* monel metal is suitable. Cast iron, platinum and mercury, for example, are unsuitable or undesirable for various reasons. Metals which accelerate the normally very slow reaction between alkali metals and ammonia to form amide should be avoided. Metals such as lead which form alloys with alkali metal should be avoided since the metal is to be isolated as such or after evaporation of the solution. Sodium itself is suitable if the solid metal is being made.

The portions of the containing vessel which come into contact with alkali metal solutions should be made of a material which does not catalyse the reaction forming the metal amide. Thus if a solution of alkali metal in ammonia in presence of a solution of a salt is allowed to come in contact with steel, the reaction forming metal amide may be so catalysed as to yield amide almost entirely.

The invention will be described more fully with reference to the following examples to which the invention is not limited. The drawings diagrammatically show suitable apparatus.

Example 1.

Potassium. A solution of potassium iodide in liquid anhydrous ammonia, containing not less than 0.6 gram (prefer-

ably 0.7 gram) of potassium iodide per cubic centimeter of solution, is electrolysed with a copper cathode and a potassium amalgam anode (0.05%). The solution is at or slightly below its boiling point at atmospheric pressure. Potassium is removed from the amalgam and forms at the cathode a bronze coloured solution of potassium in ammonia. This bronze liquid rises from the cathode to the surface of the iodide solution where it accumulates, and is easily drawn off continuously and without admixture of iodide solution, by means of a suitable separator which is kept at a temperature below that at which the solution boils.

The impoverished amalgam is withdrawn from the cell and replaced by fresh amalgam. If the amalgam drawn off contains 0.01% of potassium the current density at the anode may be 0.03 amp. per sq. cm.

The potassium solution may be evaporated at atmospheric pressure, which minimises the loss by reaction with ammonia to potass-amide. The residual metal is somewhat spongy; it is fused at as low a temperature as possible and run off into moulds in a pure state.

The part of the electrolytic cell in which the potassium solution collects and also the vaporator should be made of or lined with material which does not accelerate the reaction between potassium and ammonia. Ebonite, glass, enamelled iron and copper may be mentioned as examples of suitable materials.

If a solution of iodide considerably more dilute than that specified is used, no potassium is obtained, but only potassamide.

In Fig. 1 of the drawing, the electrolyte is contained in a cast iron vessel 1, the upper part of which is protected by an ebonite lining 2. Current is supplied at 3 so that the vessel is the anode and a perforated steel plate 4 serves as the cathode, this is insulated at 5. The amalgam flows in at 6 and out at 7. Liquid anhydrous ammonia is supplied through valve 9 to container 8. Ammonia gas passes along pipe 10 and may be drawn off at 16. The upper liquid layer of potassium solution collects in vessel 11 overflowing into vessel 12 from which it is withdrawn through pipe 14, vessel 15 and valve 17. The iodide solution overflowing with the potassium solution is returned through pipe 13. The apparatus may be under pressure if desired. The cooling jackets (necessary especially for vessel 12) are not illustrated.

Example 2.

Solid sodium. A solution of 90 to 100 grams sodium cyanide in 100 grams of liquid ammonia is electrolysed at atmospheric pressure and at temperatures between its boiling and freezing points (approximately between $-20°$ C. and $-34°$ C) with a mild steel cathode and sodium amalgam (0.05%) anode. Sodium is deposited in the form of a solid crystalline sponge which is collected together by any suitable means within the cell such as a knife and piston and without exposure to air or moisture and then compressed in order to expel the enclosed electrolyte and cause the separate crystals to coalesce into a mass of homogeneous metal. This metal is then extruded by pressure through an orifice in the cell wall which is always closed by the plug of solid sodium. The current efficiency in this case is almost theoretical.

The process may be performed in the apparatus very diagrammatically shown in Fig. 2 which is not part of the invention covered by this application.

In this drawing, 20 is a vessel (cooled by a jacket not shown) 21 is a slowly rotating cathode drum, 22 is the amalgam, 24 a reciprocating piston, 25 the knife and 26 the solid sodium extruded through outlet 27.

In this application I do not claim the removing of the metal sodium from sodium amalgam claimed in my Specification No. 218,373.

Dated this 7th day of December, 1923.

W. P. THOMPSON & Co.,
12, Church Street, Liverpool,
Chartered & Registered Patent Agents.

COMPLETE SPECIFICATION.

Improvements in and relating to the Manufacture of Alkali Metals.

I, (Dr.) Thomas Ewan, a British subject, of 86, Shuna Street, Maryhill, Glasgow, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to an improve-

ment in the manufacture of alkali metals.

According to my invention I electrolyse an aqueous solution of an alkali salt with a mercury cathode and I remove the alkali metal from the amalgam by the aid of liquid anhydrous ammonia. My invention further consists in the process of separating sodium (or other alkali metal) as such from amalgam especially from a very dilute amalgam by using the amalgam as anode in the electrolysis of an inert electrolytic solution at low temperature *i.e.* at temperatures much lower than 100° C. The preferred range is from − 30° C. to + 30° C. which is far below those which can be obtained by the use of fused salts. The solvent I prefer to use is liquid anhydrous ammonia but the invention is not limited thereto as other solvents may be used provided that they yield electrolytic solutions inert to sodium or other alkali metal, that is, the solutions must not react with the sodium or alkali metal to an extent which will prevent isolation of the metal therefrom. Liquid methylamine is an example of an alternative solvent. Many organic liquid are excluded because they react with sodium or alkali metal. Salts in the fused condition are unsuitable because their melting points are too high.

The electrolyte may be any sodium (or alkali-metal) salt which is soluble in the solvent and which is inert (in the above-defined sense) to sodium (or other alkali metal) for example, sodium (or other alkali metal) cyanide.

The invention includes the use of certain solutions in which alkali metals are substantially insoluble but which can be used to remove them from their amalgams. The metals can thus be separated as solids direct, but solutions in the liquefied gas may be drawn off continuously to yield the metals by evaporation. The whole process of isolation of the metals may be made continuous.

The invention also includes a suitable apparatus as defined in the claims.

My experiments lead to the conclusion that solutions of alkali metal salts in liquid anhydrous ammonia may be divided into three groups:—

(1) Those in which the corresponding metal dissolves; the solution may or may not consist of two phases.

(11) Those in which the metal is insoluble or very sparingly soluble and with which it co-exists in the form of a solution of the metal in ammonia free or nearly free from salts.

(111) Those in which the metal is substantially insoluble and with which it co-exists in the solid form.

When a readily soluble sodium salt for example, is added in increasing quantities to a solution of sodium in ammonia a point can be reached at which the solution separates into two distinct layers, the lower one consisting of a solution of the sodium salt in ammonia containing mere traces of free sodium and the upper one containing substantially the whole of the sodium dissolved in ammonia and almost free from the salt. Further increase of the concentration of the salt increases the concentration of the sodium solution in equilibrium with it until saturation is reached, beyond this point solid sodium separates out.

Solutions belonging to the first group (those in which the metal dissolves more or less readily) are of little value in the manufacture of the metals by my invention because the metal which is formed at the cathode dissolves more or less readily in the electrolyte and is reabsorbed therefrom by the mercury at the anode. Further any metal which escapes reabsorption can only be removed from the cell together with the dissolved salt from which it must be separated, in a subsequent operation, a very undesirable complication of the process. The more nearly the solutions approach the border line which divides them from group two, the less do these objections hold good. When using dilute solutions I prefer to use a diaphragm to hinder contact of dissolved sodium with mercury.

The solutions of the second group (those from which alkali metal separates almost completely in the form of a solution in ammonia) are very suitable electrolytes for the purposes of this invention. The metal solutions float on the surface of the salt solutions and are thereby effectively kept out of contact with the anode amalgam. Being liquid, they are readily removed from the electrolytic cell, and the metal is easily recovered in a pure condition by evaporating off the ammonia.

Solutions of the third group are also suitable although the deposition of solid metal, with its tendency to form dendritic crystalline growths and the removal of the solid metal from a cell, which, on account of the volatile nature of the solution is necessarily closed gas-tight present difficulties which are not met with when the metal is produced in the form of a liquid solution.

The number of sodium or alkali metal salts which yield suitable electrolytic

solutions is limited. The majority are either insoluble or so sparingly soluble in ammonia that only solutions of the first group are obtainable. Sodium chloride and bromide for example, yield solutions of this first group only.

Other salts which are sufficiently soluble cannot be used because they are not inert, *i.e.* they react with sodium or other alkali metal; the nitrates and thio cyanates being examples.

Sodium cyanide and sodium iodide are sufficiently soluble to yield solutions of groups of two and three. They do not react with sodium in presence of liquid ammonia and the solutions are good conductors of electricity but I prefer the cyanide since there is a danger of forming explosive products in certain cases when using the iodide.

The alkali metal amalgam may be of any strength, but the very dilute, liquid amalgams which are easily obtained are preferred. In using these the current density at the anode must be proportioned to the alkali metal content of the amalgam and to its rate of movement; if too high current density is employed mercury dissolves as well as alkali metal and the production of the latter is diminished. So far my experiments have indicated that (with sodium amalgam for example) an anodic current density of 3 or 4 amperes per square centimetre for each 1 *per cent.* of sodium in a stationary amalgam is safe but very much higher current densities can be employed if the amalgam is stirred or flows in a thin sheet; if these critical current densities are surpassed mercury dissolves, when the production of sodium may stop. The current density at the cathode may be quite small as compared with processes in which fused salts are electrolysed.

The cathode is suitably made of wrought iron or mild steel, but many other materials which conduct electricity and are not acted on by the substances present in the cell are available, *e.g.* monel metal is suitable. Cast iron, platinum and mercury, for example, are unsuitable or undesirable for various reasons. Metals which accelerate the normally very slow reaction between alkali metals and ammonia to form amide should be avoided. Metals such as lead which form alloys with alkali metal should be avoided since the metal is to be isolated as such or after evaporation of the solution. Sodium itself is suitable if the solid metal is being made.

The portions of the containing vessel which come into contact with alkali metal solutions should be made of a material which does not catalyse the reaction forming the metal amide. Thus if a solution of alkali metal in ammonia in presence of a solution of a salt is allowed to come in contact with steel, the reaction forming metal amide may be so catalysed as to yield amide almost entirely.

The invention will be described more fully with reference to the following examples to which the invention is not limited. The drawings left with my provisional application diagrammatically show suitable apparatus.

EXAMPLE 1.

POTASSIUM.

A solution of potassium iodide in liquid anhydrous ammonia, containing not less than 0.6 gram (preferably 0.7 gram) of potassium iodide per cubic centimeter of solution, is electrolysed with a copper cathode and a potassium amalgam anode (0.05%). The solution is at or slightly below its boiling point at atmospheric pressure. Potassium is removed from the amalgam and forms at the cathode a bronze coloured solution of potassium in ammonia. This bronze liquid rises from the cathode to the surface of the iodide solution where it accumulates, and is easily drawn off continuously and without admixture of iodide solution, by means of a suitable separator which is kept at a temperature below that at which the solution boils.

The impoverished amalgam is withdrawn from the cell and replaced by fresh amalgam. If the amalgam drawn off contains 0.01% of potassium the current density at the anode may be 0.03 amp. per sq. cm. The potassium solution may be evaporated at atmospheric pressure, which minimises the loss by reaction with ammonia to potass-amide. The residual metal is somewhat spongy; it is fused at as low a temperature as possible and run off into moulds in a pure state.

The part of the electrolytic cell in which the potassium solution collects and also the evpaorator should be made of or lined with material which does not accelerate the reaction between potassium and ammonia. Ebonite, glass, enamelled iron and copper may be mentioned as examples of suitable materials.

If a solution of iodide considerably more dilute than that specified is used, no potassium is obtained, but only potassamide.

In Fig. 1 of the drawing left with my provisional application the electrolyte is

contained in a cast iron vessel 1, the upper part of which is protected by an ebonite lining 2. Current is supplied at 3 so that the amalgam is the anode and a perforated steel plate 4 serves as the cathode;—this is insulated at 5. The amalgam flows in at 6 and out 7. Liquid anhydrous ammonia is supplied through valve 9 to container 8. Ammonia gas passes along pipe 10 and may be drawn off at 16. The upper liquid layer of potassium solution collects in vessel 11 overflowing into vessel 12 from which it is withdrawn through pipe 14, vessel 15 and valve 17. The iodide solution overflowing with the potassium solution is returned through pipe 13. The apparatus may be under pressure if desired. The cooling jackets (necessary especially for vessel 12) are not illustrated.

Example 2.

SOLID SODIUM.

A solution of 90 to 100 grams sodium cyanide in 100 grams of liquid ammonia is electrolysed at atmospheric pressure and at temperatures between its boiling and freezing points (approximately between $-20°$ C. and $-34°$ C.) with a mild steel cathode and sodium amalgam (0.05%) anode. Sodium is deposited in the form of a solid crystalline sponge which is collected together by any suitable means within the cell such as a knife and piston and without exposure to air or moisture and then compressed in order to expel the enclosed electrolyte and cause the separate crystals to coalesce into a mass of homogeneous metal. This metal is then extruded by pressure through an orifice in the cell wall which is always closed by the plug of solid sodium. The current efficiency in this case is almost theoretical.

The process may be performed in the apparatus very diagrammatically shown in Fig. 2 of the drawings left with my provisional application which is not part of the invention coveied by this application.

In this drawing, 20 is a vessel (cooled by a jacket not shown) 21 is a slowly rotating cathode drum, 22 is the amalgam, 24 a reciprocating piston, 25 the knife and 26 the solid sodium extruded through outlet 27.

In this application I do not claim the removing of the metal sodium from sodium amalgam as claimed in my Specification No. 218,373 or of the metals sodium & potassium as claimed in my Specification No. 218,606.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. The process of obtaining an alkali metal which comprises removing the alkali metal from an amalgam at a temperature much lower than 100° C. by using the amalgam as anode in an electrolytic cell in which the electrolyte is a solution of an inert salt of the alkali metal in an inert solvent and then isolating the alkali metal.

2. The process of obtaining an alkali metal which consists in transferring the metal from an amalgam to a volatile solvent by the aid of an electric current, and then evaporating the solvent to yield the metal direct.

3. The process of obtaining an alkali metal by electrolytically removing the metal from an amalgam anode by the aid of a solution of an inert salt in liquid anhydrous ammonia and then isolating the alkali metal.

4. The process as claimed in Claim 1 or 2 or 3 in which potassium is obtained.

5. The process as claimed in Claim 1 in which the composition of the solution is such that the alkali metal is substantially insoluble therein.

6. The process as claimed in Claim 1 wherein the composition of the electrolytic solution is such that two liquid layers are produced of which one contains the dissolved salt and into the other of which the alkali metal passes.

7. The process as claimed in Claim 1 in which the metal is deposited electrolytically on a suitable cathode.

8. The process of obtaining alkali metal which comprises electrolysing an aqueous solution of an alkali metal salt with a mercury cathode and recovering the alkali metal from an anode of very dilute amalgam by the aid of liquid anhydrous ammonia.

9. The process as claimed in Claim 1 in which the solution is electrolysed under superatmospheric pressure.

10. The process as claimed in Claim 1 in which the solution is electrolysed at or below its boiling point under atmospheric pressure by the aid of suitable cooling means.

11. The method of removing alkali metal from an amalgam thereof which consists in electrolysing a solution of the alkali metal cyanide in liquid anhydrous ammonia with an amalgam anode.

12. An apparatus for carrying out the process claimed in Claim 6 comprising the combination of a cell to contain, and

prevent loss of, liquid anhydrous ammonia or the like with volatile solvent whose exposed portions are made of or lined with a non-catalytic material, means to supply ammonia or the like thereto, means to supply a liquid alkali metal amalgam to form an anode, means to remove the spent amalgam, an electrical connection therefor, a kathode and means to separate an upper layer of metal solution from a lower layer of salt solution, substantially as described.

Dated this 7th day of December, 1923.

W. P. THOMPSON & Co.,
12, Church Street, Liverpool,
Chartered & Registered Patent Agents.

Hereford : Printed for His Majesty's Stationery Office, by The Hereford Times Ltd.
[Wt. 21A—125/9/1925.]

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awing is a reproduction of the Original on a reduced scale]

Fig.1.

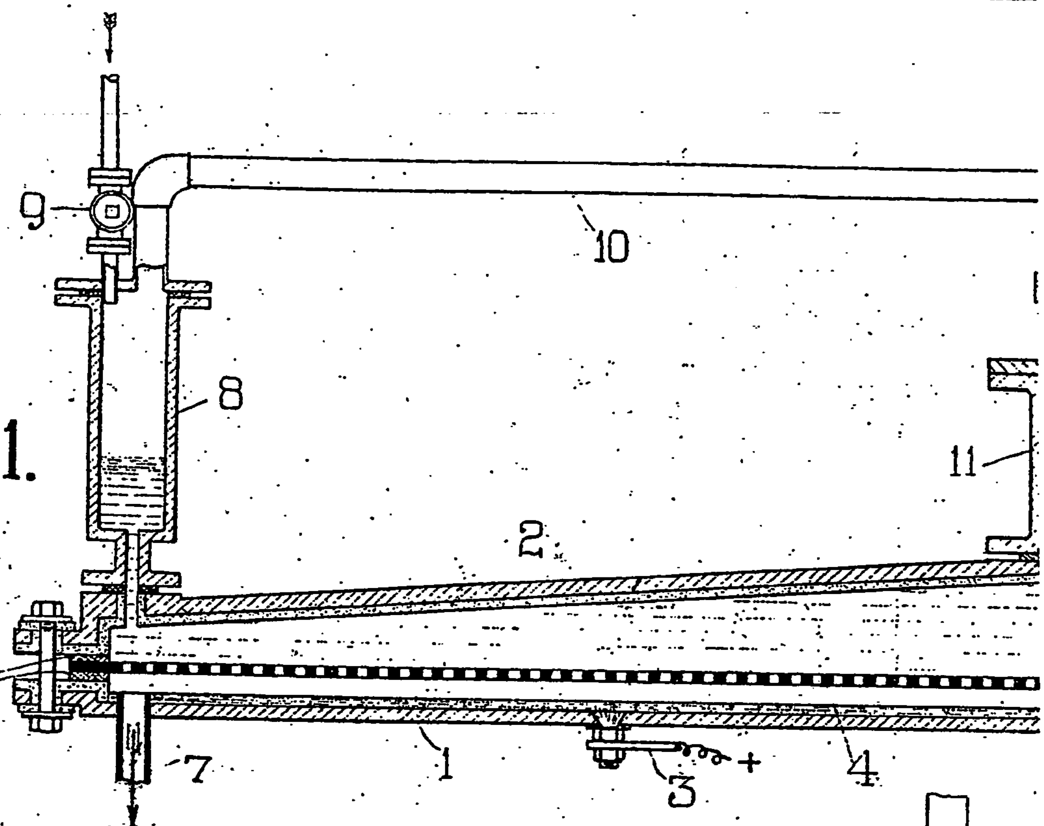
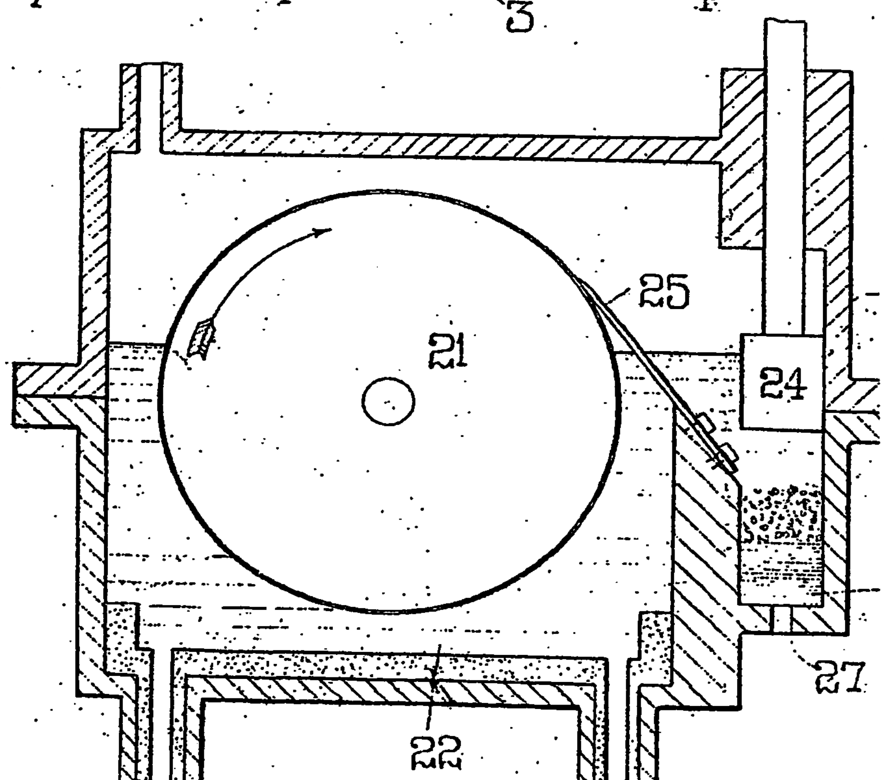


Fig.2.



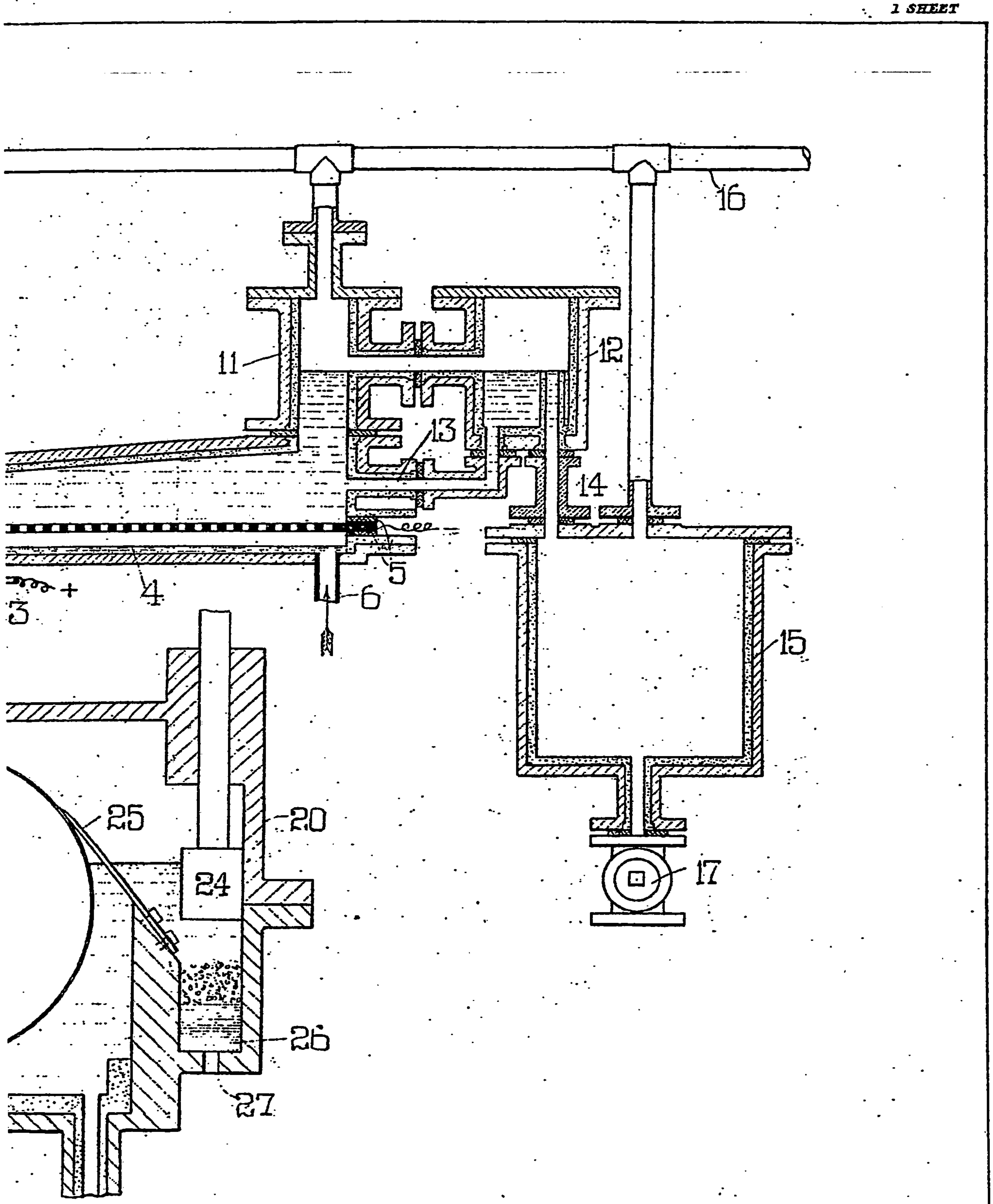


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Fig.1.

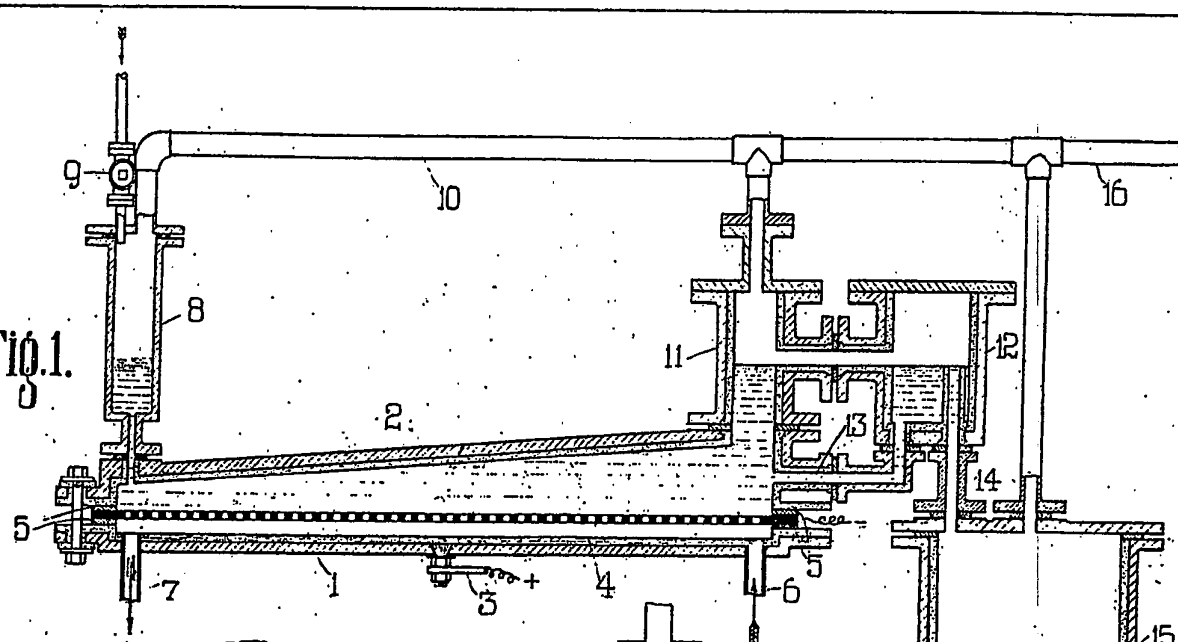
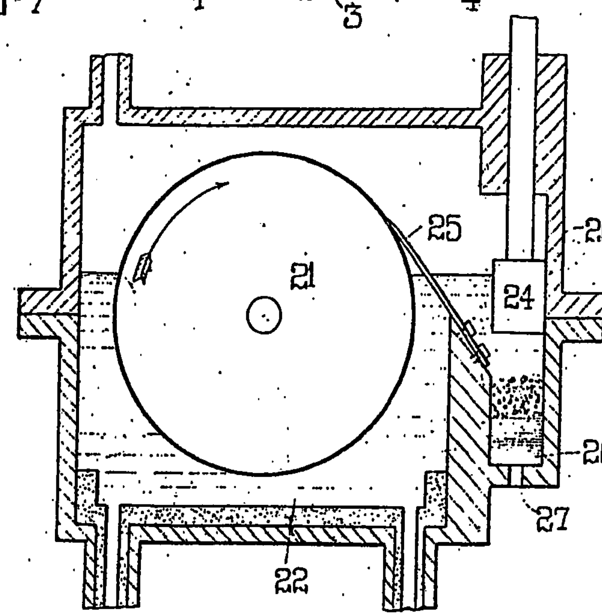


Fig.2.



[...awing is a reproduction of the Original on a reduced scale]

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